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14. ABSTRACT
In this proposal we attacked the problem of kinking nonlinearity using a number of techniques and approaches. We used nanoindentation, modeling and simple compression experiments on a variety of materials that ranged from the hexagonal metals such as Co and Mg to mica, BaTiO3, graphite to the MAX phases. In modeling we showed, in a PRB paper, for the first time, that KNE hysteresis can be well modeled/described by the powerful Preisach-Mayergoyz model that has been successfully used for many years to describe other hysteresis phenomena,

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UU	UU	UU	UU		Michel Barsoum
					19b. TELEPHONE NUMBER
					215-895-2338

Report Title

Kinking Nonlinear Elastic Solids for Load Bearing Damping and Strain Sensing Applications

ABSTRACT

In this proposal we attacked the problem of kinking nonlinearity using a number of techniques and approaches. We used nanoindentation, modeling and simple compression experiments on a variety of materials that ranged from the hexagonal metals such as Co and Mg to mica, $BaTiO_3$, graphite to the MAX phases. In modeling we showed, in a PRB paper, for the first time, that KNE hysteresis can be well modeled/described by the powerful Preisach-Mayergoyz model that has been successfully used for many years to describe other hysteresis phenomena, such as magnetism. We now routinely use our KNE model to measure the CRSS of basal dislocations in polycrystalline solids from a simple compression experiment – another first - and showed it to follow a Hall-Petch relationship. With colleagues at Florida Institute of Technology, we developed a powerful nanoscale continuum calculation of basal dislocation core structures in graphite. This work is the first step towards developing a robust model for, first, kink boundaries and ultimately incipient kink bands. We also developed - and filed for patents for - Mg/Ti_2AlC composites with exceptional combinations of strengths, stiffness, machinability and damping. In an important paper to geologists we showed that not only is mica a KNE solid, but as important its KNE response is a function of its defect concentration.

List of papers submitted or published that acknowledge ARO support during this reporting period. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

1. B. Yang, M. W. Barsoum and R. M. Rethinam, "Nanoscale Continuum Calculation of Basal Dislocation Core Structures in Graphite", Phil. Mag., in print.
2. A. Zhou, D. Brown, S. Vogel, O. Yeheskel and M. W. Barsoum, “On the Kinking Nonlinear Elastic Deformation of Polycrystalline Cobalt”, Mater. Sci. Engin. A, 527, 4664-4673 (2010).
3. S. Amini and M. W. Barsoum, “On the Effect of Texture on the Mechanical Properties of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases”, Mater. Sci. Engin. A, 527, 3707-3718 (2010).
4. A. Zhou, S. Basu, P. Finkel, G. Friedman and M. W. Barsoum, “Hysteresis in Kinking Nonlinear Elastic Solids and the Preisach-Mayergoyz Model”, Phys. Rev. B, 82, 094105 (10 pp) (2010).
5. A. G. Zhou and M. W. Barsoum, "Kinking Nonlinear Elastic Deformation of Ti_3AlC_2, Ti_2AlC,$Ti_3Al(C_{0.5},N_{0.5})_2$ and $Ti_2Al(C_{0.5},N_{0.5})$", J. Alloys Compds., 498, 62-67 (2010).
6. F. Barcelo, S. Doriot, T. Cozzika, M. Le Flem, J.-L. Béchade, M. Radovic and M. W. Barsoum, “Electron-Backscattered Diffraction and Transmission Electron Microscopy Microstructural Study of Post-Creep Ti_3SiC_2”, J. Alloy Compds., 488, 181–18 (2010).
7. P. Finkel, A.G. Zhou, S. Basu, O. Yeheskel & M. W. Barsoum, "Direct Observation of Nonlinear Acousto-Elastic Hysteresis in Kinking Nonlinear Elastic Solids”, Appl. Phys. Lett., 94,241904 (2009).
8. A. Zhou and M. W. Barsoum, “Kinking Nonlinear Elasticity and the Deformation of Mg”, Met. Mater. Trans. A, 40A, 1741-1756 (2009).
9. S. Amini, J. M. Córdoba Gallego, L. Daemen, A. R. McGhie, C. Ni, M. Odén, L. Hultman & M. W.Barsoum, “On the Stability of Mg Nanograins to Coarsening after Repeated Melting”, Nano Letters, 9,
3082–3086 (2009).
10. S. Basu, A. Zhou and M. W. Barsoum, “On Spherical Nano- indentations,Kinking Nonlinear Elasticity of Mica Single Crystals and Their Geological Implications”, J. Struct.Geology, 31,791–801 (2009).
11. R. Buchs, S. Basu, O. Elshrief, R. Coward and M. W. Barsoum, “Vickers and Spherical Nanoindentation Study of the Deformation of Poled $BaTiO_3$ Single Crystals”, J. Appl. Phys., 105, 093540 (2009).
12. S. Basu, M. Radovic and M. W. Barsoum, “Room Temperature Constant-Stress Creep of a Brittle Solid Studied by Spherical Nano-indentation”, J. Appl. Phys., 104, 063522 (2008).

Number of Papers published in peer-reviewed journals: 12.00

(b) Papers published in non-peer-reviewed journals or in conference proceedings (N/A for none)

Number of Papers published in non peer-reviewed journals: 0.00

(c) Presentations

Invited Talks:
1) PLENARY lecture: 7th Inter. Conf. on High Temp. Ceram. Matrix Comp., Bayreuth, Germany, Sept. 2010.
2) Paul Schererr Institute, Zurich, Switzerland, July 2010.
3) EMPA, Thun, Switzerland, July 2010
4) CIMTEC, Montecatini, Italy, June, 2010.
5) KEYNOTE lecture: 1st Intern. Conf. on Materials for Energy 2010, Karlsruhe, Germany, July 2010.
6) Harbin Institute of Technology, Harbin, China, May 2010.
7) Harbin Institute of Technology, Harbin, China, May 2010.
8) CAMTEC II, Cambridge, England, March 2010; NI/KNE talk.
9) 34th ICACC in Daytona Beach, FL, January 2010.
10) Lake Louise Conference, Lake Louise, Canada, Oct. 2009.
11) TM&S Fall Meeting, Pittsburgh, PA, Oct. 2009.
12) MS&T'09 Fall Meeting, Pittsburgh, Oct. 2009.
13) Aachen University, Aachen, Germany; Sept. 2009.
14) Uppsala Univ., Uppsala, Sweden, May 2009.
15) Int. Conf. Metal. Coating & Thin Films. San Diego, CA, April 2009.
16) Linkoping University, Linkoping, Sweden, Nov. 2008.
17) MS&T, Pittsburgh, PA, Oct. 2008.
18) E-MRS Fall Meeting, Warsaw, Poland, Sept. 2008.
19) Dupont Experimental Station, Delaware, March 2008; MAX/KNE talk.
20) Plenary Lecture, 13th Israeli Maters Engin. Conf., Dec. 2007

Regular Talks (given by students).

21) 2010- MS&T Conference, Houston, TX. Mechanical Properties and Kinking Non-Linear Elasticity of Nano-Crystalline Mg-Matrix Composites Reinforced with MAX Phases.
22) 2010- MS&T Conference, Houston, TX. Thermal Stability of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases
23) 2010- NATAS Annual Conference, Philadelphia, PA. Thermal Stability of Nanocrystallin Mg-matrix Composites reinforced with MAX Phases
24) 2010- TAFDV Spring Symposium, Philadelphia, PA, On the Stability of Mg Nanograins to Coarsening after Repeated Melting
25) North American Thermal Analysis Society Annual Conf.,Lubbock, TX: On the Thermal Stability of Nanocrystalline Mg in Mg-matrix Composites reinforced with MAX Phases
26) 7th Pacific Rim International Conference on Advanced Materials and Processing,1-6 August 2010, Cairns, Australia: Thermal Stability and Effect of Texture on Ultrahigh Damping of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases.
27) MS&T 2010 Conference and Exhibition, Houston, TX: On the Thermal Stability of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases.
28) MS&T 2010 Conference and Exhibition, Houston, TX: Mechanical Properties and Kinking Non-Linear Elasticity of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases.
29) MS&T 2009 Conference and Exhibition, Pittsburgh, PA: Thermal Stability and Effect of Texture on Ultrahigh Damping of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases.
30) MS&T 2009 Pittsburgh, PA: Mechanical Properties and Kinking Non-Linear Elasticity of Fully Dense Ti2SC and Cr2GeC.
31) MS&T 2009, Pittsburgh, PA: Thermal Expansion of Select MAX Phases Measured by High Temperature X-ray Diffraction and Dilatometry.
32) MS&T 2009, Pittsburgh, PA: Microstructural Characterization and Mechanical Properties of a Ti2AlC/Nanocrystalline Mg-Matrix Composite.
33) TMS 2009, San Francisco, CA: Processing, Microstructural Characterization and Mechanical Properties of a Ti2AlC/Nano- crystalline Mg-Matrix Composite.
34) ACERS 2009, Int. Conference & Exposition on Advanced Ceramics Daytona Beach, FL: Mg Matrix Composites Reinforced with MAX Phases.
35) ACERS 2009, International Conference & Exposition on Advanced Ceramics & Composites, Daytona Beach, FL: Mechanical Properties and Ultrahigh Damping of Oriented MAX Phases.
36) MS&T 2008 Conference and Exhibition, Pittsburgh, PA: Ti2AlC/Nanocrystalline Mg-Matrix Composites.

Upcoming
37) 2011- ACERS Conference, Daytona Beach, FL. Thermal Stability and Effect of Texture on Ultrahigh Damping of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases.
38) 2011- ACERS Conference, Daytona Beach, FL. Isothermal Oxidation of Ti2GeC in Air.
39) 2011- TMS Annual Conference, San Diego, CA. Nanocrystalline Mg-Matrix Composites with Ultrahigh Damping Properties.
40) 2011- TMS Annual Conference, San Diego, CA. Effect of Reinforcement Volume Fraction on the Properties of Nc-Mg Matrix Composite Reinforced with Ti2AlC.

Number of Presentations: 40.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

B. Anasori, S. Amini, V. Presser and M. W. Barsoum, "Nanocrystalline Mg-Matrix Composites with Ultrahigh Damping Properties" TMS Conference 2011, Proceeding Title: Mg Technology 2011, San Diego, CA.

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts): 1

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts): 0

(d) Manuscripts

Number of Manuscripts: 0.00

Patents Submitted

1) MAX-Based Metal Matrix Composites

Patents Awarded

1) A Method for Structural Health Monitoring Using a Smart Sensor System

Awards

1) ISI's Most Highly Cited Authors List 2009 (http://isihighlycited.com/).
2)Visiting Professor, Linkoping University, Linkoping, Sweden.
3) A. W. Grosvenor Professor, Department of Materials Science and Engineering, 2009.
4) Wheatly Scholar, Los Alamos National Laboratory, Los Alamos, NM, October 2008.
5) 2008 Sigma Xi Lecture, MIT, Cambridge, MA, May 2008.
6) Outstanding Research Award, Department of Materials Science and Engineering, 2008.
7) University Research/Scholarship Award, Drexel University, 2007 (Inaugural award).

Graduate Students

NAME	PERCENT_SUPPORTED
Sandip Basu	0.50
Babak Anasori	1.00
Shahram Amini	0.50
FTE Equivalent:	**2.00**
Total Number:	**3**

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Michel Barsoum	0.20	No
FTE Equivalent:	**0.20**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME	
Shahram Amini	
Sandip Basu	
Total Number:	**3**

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Summary of Our Most Notable Accomplishments to Date:

1) Composites with record values of energy dissipated per cycle per unit volume

In previous reports and papers we showed that the MAX phases, more specifically, Ti2AlC and the hexagonal metals, including Mg, were both kinking nonlinear solids. This led us to postulate that Mg composites reinforced with Ti2AlC should result in solids with large energies dissipated per cycle per unit volume, Wd, as a result of the formation and annihilation of dislocation-based incipient kink bands, IKBs. In a recent paper, we reported on the processing and microstructural characterization of 50 vol. % Ti2AlC/nanocrystalline (nc) Mg-matrix composites fabricated by pressureless melt infiltration at 750 °C for 1 h. X-ray diffraction and transmission electron microscopy both confirmed that the Mg grain size was ~35±15 nm. Some Mg was dissolved in the Ti2AlC confirming the existence of a (Ti1-xMgx)2AlC solid solution, with x as high as 0.2. A small amount of Ti (3±1 at. %) was also found in the Mg-matrix. In a paper published in Nano Letters, we showed that this microstructure was also exceptionally stable; annealing for 6 h at 550 °C did not alter the size of the Mg-grains. More recently we showed that heating this nano-grained Mg at 500 °C for 8h had absolutely no effect on the mechanical properties of the composite, confirming the extraordinary thermal stability of the Mg-matrix.

At 350±40 the ultimate tensile strength is significantly greater than other pure Mg-composites reported in the literature. At 700±10 MPa, the ultimate compressive stresses of these composites were ˜ 40% higher than those of a 50 vol.% Ti3SiC2-Mg or a 50 vol.% SiC-Mg, in which the Mg-matrix grains were not at the nanoscale. The Ti2AlC/nc-Mg composites are readily machinable, stiff (˜ 70 GPa), strong, light (2.9 g/cm3) and exhibited exceptional damping capabilities, that increased as the square of the applied stress to stress levels of the order of ˜ 500 MPa. The Wd values at such stress levels was, for a short time (see below) the highest ever reported for a crystalline solid. This work resulted in a patent application by Drexel University. Thixomat of Ann Arbor, MI is also testing these composites for armor applications.

2) Effect of Texture on Energy dissipated per cycle.

Given that kinking is a plastic instability, it was further postulated that if the Ti2AlC grains were aligned such that their basal planes were parallel the direction of the applied stress then kinking would be more prevalent, which in turn should result in even higher Wd values than if there were no texture. To test this idea we fabricated and tested the effects of texture on KNE and Wd. At 450 MPa, Wd of the composites in which the Ti2AlC grains were oriented parallel to the applied load direction – i.e. edge on - texture was found to be ˜ 0.6 MJ/m3, a value that exceeds our previous record (see above) of ˜ 0.4 MJ/m3 at 475 MPa reported for a randomly oriented composite. The main role of the nc-Mg matrix was to enhance the ultimate compressive and tensile stresses of the composites. At 700±10 and 380±20 MPa, the ultimate compressive and tensile strengths of the composites were significantly higher than those reported in the literature for composites in general and in which the matrices were pure Mg, in particular. This paper was submitted for publication.

Because all composite and bulk Ti2AlC samples tested traced fully reversible, reproducible, hysteretic loops during uniaxial cyclic compression tests, they were classified as KNE solids. When the results were analyzed using our recently developed microscale IKB-based model, the various relationships predicted among the three independently measured values – stress, nonlinear strain and Wd – were exceptionally well adhered to. In all composites, and despite very different loop shapes and sizes, the critical resolved shear stresses of basal plane dislocations in Ti2AlC, calculated from the model, fell within the narrow range of 37.7±0.5 MPa. The same was true for the reversible dislocation density that fell in the quite narrow range of 1.1±0.3×1014 m-2, suggesting the presence of an equilibrium state to which all the systems migrate.

3) Hysteresis in Kinking Nonlinear Elastic Solids and the Preisach-Mayergoyz Model

In a paper published in PRB, we show that the stress-induced, dislocation-based, elastic hysteric loops of kinking nonlinear elastic solids – polycrystalline cobalt, 10 vol. % porous Ti2AlC and fully dense Ti3SiC2– obey the scalar Preisach-Mayergoyz phenomenological model because they exhibit wipe-out and congruency, two necessary and sufficient tenets of the model. We also demonstrate the power of the model in predicting the response of these materials to complex stress histories, as well as, determining the distributions of the threshold and friction stresses associated with the incipient kink bands – the fundamental microscopic units responsible for kinking non-linear elasticity.

4) Encyclopedia Entry on Kinking Non-linear Elastic Solids

The third entry by the PI of this proposal in the Encyclopedia of Materials Science and Technology, deals with KNE solids. The first 2 entries were on the MAX phases - KNE solids are ubiquitous in nature and span the gamut of bonding in solids, from metallic, to covalent, to ionic and van der Waals and combinations thereof. Plastic anisotropy is a sufficient condition for belonging to the set. IKBs, the precursors of regular KBs, are the micro-mechanism responsible for kinking nonlinear elasticity. The nucleation and growth of IKBs are believed to be responsible for the fully, reversible, strain rate independent – at least up to 10-3 s-1 - stress-strain curves that close spontaneously.

The following factors have been shown to affect the size and shapes of the reversible loops:

a) Grain size; the larger the grain size, the larger 2a and hence the lower the threshold stresses, which, for a given s, in turn results in larger stress-strain loops and larger Wd values. The size to which the dislocation loops can grow is also larger.

b) Shear moduli: The lower the values of G, the larger Wd. This is a strong factor since Wd is inversely proportional to G. This factor also explains why, at a given stress, porous solids can dissipate more energy than fully dense solids.

c) CRSS or O/b: This is an important factor and is essentially a measure of the CRSS of IKB dislocations. All else being equal, solids with higher CRSS will dissipate more energy than ones that have lower values. This comment notwithstanding, since there is a strong correlation between CRSS and the threshold stresses, the net result of having solids with high values of CRSS to enhance Wd has to be considered on a case-by-case basis.

d) Dislocation core width, w: In principle this factor is related to the critical kinking angle, ¿c and the narrower the dislocations, the higher ¿c, and the lower the values of Wd. Since to date all dislocations were assumed to have the same w, this factor remains to be tested.

e) Texture: Texture can and will affect the Taylor factor, M, which as noted above can play an important role.

5) In a paper published in J. Structural Geology, we show, using cyclic spherical nanoindentation experiments, that the deformation mechanisms in mica, including basal plane ruptures and delaminations, can be explained by invoking the presence of mobile dislocation walls, incipient and regular kink bands. Our results clearly show that the energy dissipated, or that stored, during the deformation of muscovite depends critically on its previous deformation history and/or the pre-existing defect concentration. Once nucleated, the dislocation-based incipient kink bands are believed to be responsible for the nonlinear elastic deformation and hysteretic loops obtained during cyclic loading. Moreover, a model by which the number and distribution of dislocations under the indenter can be estimated and the energy consumed in their motion is presented. From the model, we also estimate the critical resolved shear stress for the motion of basal plane dislocations under an indenter. The implications of this work can be extended beyond mica to understand the nonlinear hysteretic deformation in other geological formations dominated by layered minerals.

6) In a paper published in J. Alloys and Compounds, four ternary carbide phases, Ti_3AlC_2, Ti_2AlC, $Ti_3Al(C_{0.5},N_{0.5})_2$ and $Ti_2Al(C_{0.5},N_{0.5})$, were fabricated and subjected to cyclic loading. Not surprisingly, they are all KNE solids. When the results were analyzed using our IKB model, the various relationships predicted between the three independently measured and calculated values - stress, energy dissipated per unit volume per cycle and nonlinear strain - were exceptionally well adhered to. Interestingly, the results showed that grain size played a more important role in determining the CRSS than chemistry. Lastly we note that the fact that we now can – using our IKB model – routinely calculate the CRSS of the dominant slip system from a simple cyclic compression experiment of a polycrystalline sample will prove to be very useful for metallurgists and material scientists since its will help elucidate the relationships between the yield points and CRSS. Such a measurements was not possible to date.

7) In a very recent paper published in Phil. Mag. work, we calculate the core structures of basal dislocations in graphite in a nanoscale continuum framework. The model consists of a stack of buffered Kirchhoff plates where the plates represent the covalent interactions within individual graphene sheets and the buffer layers represent the secondary interactions between them. In the mid-plane of the buffer layers, cohesive surfaces are introduced to account for the nonlinear deformations due to basal dislocations. The cohesive surface separation is governed by using an empirical 4-8 Lennard-Jones potential. Meanwhile, their relative shear sliding is governed by using a newly proposed empirical periodic stacking-fault potential. With these potentials, the core structures of full and partial dislocations are calculated and examined. We show how full dislocations automatically split into partials that repel each other. The core size of each partial, measured between peak stresses, is about 5 nm wide for the edge component and slightly narrower for the screw component. Since this size is about 10 times the lattice constant, it lends credence to our continuum model of basal dislocation cores in graphite. We also show that when the dislocations are densely packed on the same glide plane, i.e., in a pile up, with spacing one to two times the core size, the split partials retain their individual identity with well-defined and well-separated stress peaks. Meanwhile, the membrane normal stresses in the graphene sheets rise considerably at the pile-up tips, which in turn may explain further deformation mechanisms in graphite such as kinking and delamination.

8) In a paper in Appl. Phys. Letters, we reported on direct experimental observation of nonlinear hysteretic ultrasonic wave transmission through the kinking nonlinear elastic solids, Ti_3SiC_2 and Ti_3AlC_2 under bias stress loading. We observed two characteristic regimes: Up to strain ˜ $2x10^{-4}$, the ultrasound attenuation increased strongly and linearly with strain. At higher strains, the attenuation was fully reversible and hysteretic as the compressive stresses were cycled. This hysteretic behavior was attributed to interaction of the acoustic waves with dislocations in the incipient kink bands, the micro-mechanism believed to be responsible for the concomitant hysteretic stress-strain loops. This work led to the filing of a patent titled: “Smart Sensor Based on Nonlinear Elasticity and Ultrasound Attenuation”.

Technology Transfer